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DB=USPT; PLUR=YES; OP=ADJ

<u>L30</u>	L29 and skin	3	<u>L30</u>
<u>L29</u>	L28 and l1	17	<u>L29</u>
<u>L28</u>	L27 and membrane	87	<u>L28</u>
<u>L27</u>	L26 and copolymer	96	<u>L27</u>
<u>L26</u>	L25 and tetrafluoroethylene	99	<u>L26</u>
<u>L25</u>	hexafluoropropylene and hollow fiber	120	<u>L25</u>
<u>L24</u>	L23 and hollow fibers	1	<u>L24</u>
<u>L23</u>	L22 and hydrophobic	22	<u>L23</u>
<u>L22</u>	L21 and asymmetric	38	<u>L22</u>
<u>L21</u>	L20 and membranes	314	<u>L21</u>
<u>L20</u>	thermoplastic same polymers and perfluorinated	1036	<u>L20</u>
<u>L19</u>	L18 and hydrophobic	3	<u>L19</u>
<u>L18</u>	L17 and l1	7	<u>L18</u>
<u>L17</u>	L16 and hollow fiber	126	<u>L17</u>

<u>L16</u>	perfluorinated same polymers and membranes	1201	<u>L16</u>
<u>L15</u>	2468664.pn.	1	<u>L15</u>
<u>L14</u>	3445434.pn.	1	<u>L14</u>
<u>L13</u>	3615024.pn.	1	<u>L13</u>
<u>L12</u>	4049589.pn.	1	<u>L12</u>
<u>L11</u>	4220543.pn.	1	<u>L11</u>
<u>L10</u>	4238571.pn.	1	<u>L10</u>
<u>L9</u>	4248913.pn.	1	<u>L9</u>
<u>L8</u>	4376140.pn.	1	<u>L8</u>
<u>L7</u>	3615024.pn.	1	<u>L7</u>
<u>L6</u>	4377010.pn.	1	<u>L6</u>
<u>L5</u>	4384047.pn.	1	<u>L5</u>
<u>L4</u>	L1 and hydrophobic and perfluorinated	5	<u>L4</u>
<u>L3</u>	l2 and hydrophobic	3	<u>L3</u>
<u>L2</u>	L1 and hollow fiber and perfluorinated same polymer	7	<u>L2</u>
<u>L1</u>	210/500.23.ccls.	694	<u>L1</u>

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L5: Entry 2 of 3

File: USPT

Mar 17, 1992

DOCUMENT-IDENTIFIER: US 5096584 A

**** See image for Certificate of Correction ****

TITLE: Spiral-wound membrane separation device with feed and permeate/sweep fluid flow control

Detailed Description Text (27):

In one embodiment, the single elongated membrane sheet is prepared by casting a suitable membrane solution upon a permeable polymeric base cloth and processed under conditions well known in the art to form an asymmetric membrane. This embodiment is illustrated diagrammatically in FIG. 3. Optionally, the membrane may be a composite having a relatively thin active layer, and a thicker porous membrane support 41 which is often intermeshed with the polymeric base cloth. While the two layers are shown for illustration purposes as separate, they are often in reality an integral structure and the active layer 41 is much thinner than illustrated.

Detailed Description Text (28):

The permselective membrane can be homogeneous (having a thickness of between about 10-200 microns) and having first and second surfaces. The permselective membrane can also be asymmetric having one active surface and a surface opposite to the active surface.

Detailed Description Text (68):

Non-ionic (thermoplastic) forms of perfluorinated polymers described in the following patents are particularly suitable for use in the present invention because they are easily softened by heating and formed into useful membrane shapes. Membranes which are suitable are described in the following U.S. Pat. Nos.: 3,282,875; 3,909,378; 4,025,405; 4,065,366; 4,116,888; 4,123,336; 4,126,588; 4,151,052; 4,176,215; 4,176,215; 4,192,725; 4,209,635; 4,212,713; 4,251,333; 4,270,996; 4,329,435; 4,330,654; 4,337,137; 4,337,211; 4,340,680; 4,357,218; 4,358,412; 4,358,545; 4,417,969; 4,462,877; 4,470,889; 4,478,695; and European Patent Application 0,027,009, all of which are specifically incorporated herein by reference. Such polymers usually have equivalent weight in the range of from about 500 to about 2000. The membranes can be of a single layer or they can be a multilayer membrane.

4,846,977

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4,846,977

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☐ 1. Document ID: US 6582496 B1

L3: Entry 1 of 2

File: USPT

Jun 24, 2003

US-PAT-NO: 6582496

DOCUMENT-IDENTIFIER: US 6582496 B1

TITLE: Hollow fiber membrane contactor

DATE-ISSUED: June 24, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cheng; Kwok-Shun	Nashua	NH		
Doh; Cha P.	Sudbury	MA		

US-CL-CURRENT: 95/46; 95/45, 96/14, 96/6, 96/8

Full	Title	Citation	Front	Review	Classification	Data	Reference			Claims	KMAC	Draw De
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☐ 2. Document ID: US 5034126 A

L3: Entry 2 of 2

File: USPT

Jul 23, 1991

US-PAT-NO: 5034126

DOCUMENT-IDENTIFIER: US 5034126 A

**** See image for Certificate of Correction ****

TITLE: Counter current dual-flow spiral wound dual-pipe membrane separation

DATE-ISSUED: July 23, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Reddy; Damoder	San Ramon	CA		
Moon; Tag Y.	Worthington	OH		
Reineke, deceased; Charles E.	lat of Midland	MI		

US-CL-CURRENT: 210/321.74; 210/321.83

Full	Title	Citation	Front	Review	Classification	Data	Reference			Claims	KMAC	Draw De
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☐ 1. Document ID: US 6582496 B1

L5: Entry 1 of 3

File: USPT

Jun 24, 2003

US-PAT-NO: 6582496

DOCUMENT-IDENTIFIER: US 6582496 B1

TITLE: Hollow fiber membrane contactor

DATE-ISSUED: June 24, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Doh; Cha P.	Sudbury	MA		

US-CL-CURRENT: 95/46; 95/45, 96/14, 96/6, 96/8

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KMC	Draw De
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☐ 2. Document ID: US 5096584 A

L5: Entry 2 of 3

File: USPT

Mar 17, 1992

US-PAT-NO: 5096584

DOCUMENT-IDENTIFIER: US 5096584 A

**** See image for Certificate of Correction ****

TITLE: Spiral-wound membrane separation device with feed and permeate/sweep fluid flow control

DATE-ISSUED: March 17, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Reddy; Damoder	San Ramone	CA		
Denslow; Keith	Midland	MI		

US-CL-CURRENT: 210/321.74; 210/321.83, 210/497.1

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KMC	Draw De
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☐ 3. Document ID: US 5034126 A

L5: Entry 3 of 3

File: USPT

Jul 23, 1991

US-PAT-NO: 5034126

DOCUMENT-IDENTIFIER: US 5034126 A

**** See image for Certificate of Correction ****

TITLE: Counter current dual-flow spiral wound dual-pipe membrane separation

DATE-ISSUED: July 23, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Reddy; Damoder	San Ramon	CA		
Moon; Tag Y.	Worthington	OH		
Reineke, deceased; Charles E.	lat of Midland	MI		

US-CL-CURRENT: 210/321.74; 210/321.83

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	IMC	Draw. De
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asymmetric same membrane and L1	3

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<u>L4</u>	L3 and asymmetric	2	<u>L4</u>
<u>L3</u>	L2 and hydrophobic	2	<u>L3</u>
<u>L2</u>	L1 and ultrafiltration same membrane	9	<u>L2</u>
<u>L1</u>	perfluorinated same polymer same thermoplastic	230	<u>L1</u>

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International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : B01D 69/00	A2	(11) International Publication Number: WO 00/44482 (43) International Publication Date: 3 August 2000 (03.08.00)
<p>(21) International Application Number: PCT/US00/02194</p> <p>(22) International Filing Date: 27 January 2000 (27.01.00)</p> <p>(30) Priority Data: 60/117,854 29 January 1999 (29.01.99) US</p> <p>(63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application US 60/117,854 (CIP) Filed on 29 January 1999 (29.01.99)</p> <p>(71) Applicant (for all designated States except US): MILLIPORE CORPORATION [US/US]; 80 Ashby Road, Bedford, MA 01730 (US).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): CHENG, Kwok-Shun [US/US]; 7 Federal Hill Road, Nashua, NH 03062 (US). GATES, T., Dean [US/US]; 27 Nellian Way, Bedford, MA 01730 (US). YEN, Larry, Y. [US/US]; 10 Pomeroy Road, Andover, MA 01810 (US). PATEL, Rajnikant, B. [IN/US]; 22 Breckenridge Road, Tewksbury, MA 01876 (US).</p>		<p>(74) Agent: HUBBARD, John, Dana; Millipore Corporation, 80 Ashby Road, Bedford, MA 01730 (US).</p> <p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>Without international search report and to be republished upon receipt of that report.</i></p>
(54) Title: SKINNED HOLLOW FIBER MEMBRANE AND METHOD OF MANUFACTURE		
<p>(57) Abstract</p> <p>ABSTRACT OF THE DISCLOSURE</p> <p>Hollow fiber membranes having a skinned surface on one diameter, and a porous surface on the opposite diameter are produced from perfluorinated thermoplastic polymers by extruding a heated solution of the polymer having a lower critical solution temperature directly into a cooling bath to form the porous membrane by liquid-liquid phase separation. Extrusion can be conducted either vertically or horizontally. The hollow fiber membranes are useful as ultrafiltration membranes and as membrane contactors.</p>		

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L1 and hollow fiber and perfluorinated same polymer	7

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L1 210/500.23.ccls.

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 694 L1

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<u>L23</u>	L22 and hydrophobic
<u>L22</u>	L21 and asymmetric
<u>L21</u>	L20 and membranes
<u>L20</u>	thermoplastic same polymers and perfluorinated
<u>L19</u>	L18 and hydrophobic
<u>L18</u>	L17 and l1
<u>L17</u>	L16 and hollow fiber
<u>L16</u>	perfluorinated same polymers and membranes
<u>L15</u>	2468664.pn.
<u>L14</u>	3445434.pn.
<u>L13</u>	3615024.pn.
<u>L12</u>	4049589.pn.
<u>L11</u>	4220543.pn.

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3	<u>L19</u>
7	<u>L18</u>
126	<u>L17</u>
1201	<u>L16</u>
1	<u>L15</u>
1	<u>L14</u>
1	<u>L13</u>
1	<u>L12</u>
1	<u>L11</u>

<u>L10</u>	4238571.pn.	1	<u>L10</u>
<u>L9</u>	4248913.pn.	1	<u>L9</u>
<u>L8</u>	4376140.pn.	1	<u>L8</u>
<u>L7</u>	3615024.pn.	1	<u>L7</u>
<u>L6</u>	4377010.pn.	1	<u>L6</u>
<u>L5</u>	4384047.pn.	1	<u>L5</u>
<u>L4</u>	L1 and hydrophobic and perfluorinated	5	<u>L4</u>
<u>L3</u>	l2 and hydrophobic	3	<u>L3</u>
<u>L2</u>	L1 and hollow fiber and perfluorinated same polymer	7	<u>L2</u>
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L30: Entry 1 of 3

File: USPT

Dec 17, 2002

DOCUMENT-IDENTIFIER: US 6495041 B2

TITLE: Method for purifying aqueous suspension

Abstract Text (1):

A method and apparatus for purifying an aqueous suspension comprising feeding an aqueous suspension containing fine particles comprising an inorganic component from the outer surface of a wavy hollow fiber membrane having an outer diameter of from 0.5 to 3.1 mm to filter, followed by physical wash of the hollow fiber membrane. The purifying method can reduce the damage of the membrane outer surface during the physical wash step, prevent open pores on the surface from covering and achieve stable filtration. The hollow fiber membrane bundle can be produced by having a pulsation flow contacted with the hollow fiber material being extruded from the double spinning nozzle under specific conditions and cooling and solidifying or coagulating it while shaking.

Brief Summary Text (3):

The present invention relates to a method for purifying an aqueous suspension with a module comprising wavy hollow fiber membranes. Particularly, the present invention relates to a method for purifying an aqueous suspension, for example, tap water such as river water, lake water, marsh water and ground water; water for industrial use; waste water; secondary treatment waste water; industrial sewage; domestic sewage; human waste; sea water and the like with the module.

Brief Summary Text (4):

Further, the present invention is also directed to a bundle of wavy hollow fiber membranes, a process for producing the same, and a hollow fiber membrane module comprising the same.

Brief Summary Text (6):

Heretofore, various methods for purifying aqueous suspensions such as tap water, water for industrial use, waste water, industrial sewage, domestic sewage, human waste, sea water and the like with a hollow fiber membrane have been known. In particular, a purifying method according to so-called external pressure filtration, in which raw water permeates in the direction from the outer to inner surface of a hollow fiber membrane, can secure a larger membrane area contributing to filtration per unit volume when compared to so-called internal pressure filtration, in which raw water permeates in the direction from the inner to outer surface of a hollow fiber membrane. Therefore external pressure filtration is advantageously used in the field wherein minimization of water production cost is required, for example, a water-treatment field such as turbidity removal for waterworks.

Brief Summary Text (7):

In the above method for purifying aqueous suspensions with hollow fiber membranes, suspended or organic materials having a size bigger than the pore diameter of the membranes, are hindered on the membrane surface to cause a so-called concentration polarization or form a cake layer. Moreover, the organic materials in aqueous suspension clog the pores of membranes or adhere to the inner network structure of membranes. As a result, the filtration flux upon filtering the raw water is decreased to about one tenth of that upon filtering pure water. Accordingly, there have been also known purifying methods in which physical wash of membranes is

regularly practiced after the filtration in order to achieve a more stable filtration flux.

Brief Summary Text (8):

Specifically, it has been known to carry out, after a certain-term filtration, backward wash comprising feeding a part of filtrate in the reverse direction to the filtration, i.e., in the direction from the filtrate side to the raw water side (hereinafter simply referred to as back wash), air-scrubbing comprising supplying compressed gases and/or compressed air and raw water in the direction from the lower to upper part of the hollow fiber membrane module filled with water to shake fibers and discharge suspended solids accumulating among the hollow fiber membranes from the system, and the like. For example, Japanese Patent Application Laid-Open No. 60-19002 discloses a method in which a bubble generation nozzle is arranged on the side of or down the hollow fiber membranes in the hollow fiber membrane storage container, and a gas is injected therefrom together with back wash.

Brief Summary Text (9):

On the other hand, as a membrane which can be used for the above-mentioned purifying method, a reverse osmosis membrane, an ultrafiltration membrane, a microfiltration membrane, a gas separation membrane, a nanofilter, and a deairing membrane have been known. These membranes are not used alone but used in plural, i.e., in the form of a hollow fiber membrane module. The membrane module is prepared by mounting a plurality of the membranes in a module case, sealing at least one edge thereof with a thermosetting resin such as an epoxy resin, and cutting the bonded and fixed portion to open a hollow portion. Such a module is used in various fluid treatment fields, for instance, a reverse osmosis membrane module is used for desalination of sea water or brine, production of primary pure water of ultra-pure water, and concentration of fruit juice or milk; an ultrafiltration membrane module for collection of electrodeposition paints, production of pyrogen-free water, treatment of waste water, concentration of enzymes, final filtration of ultra-pure water, and turbidity removal from tap water or waste water; a microfiltration membrane module for turbidity removal from tap water or waste water, treatment of concentrated water, germ removal and purification of fermentation liquid, and fine particle removal from chemicals, a gas separation membrane module for steam removal, condensation of hydrogen, condensation or enrichment of oxygen, condensation or enrichment of nitrogen, and condensation of carbon dioxide; a nanofilter module for removal of agricultural chemicals or halogenated organic compounds; and a deairing membrane module for deairing of water and aqueous solution. The hollow fiber membranes per se have also been studied. For example, Japanese Patent Application Laid-Open No.64-22308 discloses the art using an external pressure filtration type hollow fiber membrane module wherein hollow fibers having wavy or spiral curls at least in a part thereof are mounted instead of the conventional straight hollow fiber membrane in order to prevent such a mutual clinging of the hollow fibers that hinders raw water from flowing toward the center of the module and to use almost all the hollow fibers mounted in the module for effective filtration.

Brief Summary Text (12):

In particular, we have made studies focusing on the fact that the hollow fiber membrane surface is considerably damaged when the aqueous suspension comprising fine particles containing an inorganic component is purified according to an external pressure filtration method taking a step of physical wash such as back wash and air-scrubbing.

Brief Summary Text (13):

As a result, we have found that when an inorganic component is contained in the suspended solid accumulating among hollow fiber membranes, the outer surfaces of the membranes rub against each other through the suspended solids at the physical wash step and pores on the surface of the membranes are covered with the result that the stability of the filtration operation is deteriorated. Further, we have

found that the continuation of such a phenomenon may result in the breakage of the membranes.

Brief Summary Text (14):

As the result of our extensive and intensive studies, we successfully provided, by using a bundle of hollow fiber membranes having specific waves, diameter and further bulkiness, a purifying method enabling a stable filtration, in which the damage of membrane surfaces caused by an inorganic component at the physical wash step, is unexpectedly decreased.

Brief Summary Text (15):

Moreover, in the production of a hollow fiber membrane comprising extruding membrane production raw liquid followed by cooling and then solidification or coagulation, we have also succeeded in efficiently producing a bundle having specific waves, diameter and further bulkiness as described above by contacting a pulsation flow with hollow fiber materials under specific conditions to vibrate and cooling and solidifying or coagulating the hollow fiber materials while vibrating.

Brief Summary Text (16):

Namely, it is an object of the present invention to provide a purifying method enabling stable filtration, in which, during the physical wash step following filtration of aqueous suspension, the external surfaces of hollow fiber membranes is prevented from rubbing against each other through suspended solids in the aqueous suspension containing an inorganic component, and the covering of open pores on the surface of hollow fiber membranes is hindered. It is also an object of the present invention to provide a bundle of the hollow fiber membranes which is used for the purifying method, a process for producing the bundle, and a module mounting the bundle.

Brief Summary Text (17):

It is another object of the present invention to provide a purifying method enabling a stable filtration, in which the efficiency of the physical wash to discharge suspended solids accumulating among hollow fiber membranes is improved without damaging the surface of the hollow fiber membranes. It is also another object of the present invention to provide a bundle which can be used for the purifying method, a method for producing the membrane bundle, and a module mounting the bundle.

Brief Summary Text (18):

Insufficient physical wash may cause accumulation of suspended solids in the hollow fiber membrane bundle so that the membranes cling to each other in the shape of a rod. In such a case, raw water cannot be supplied into the bundle with the result that the amount of the filtrate to be recovered is seriously decreased. Additionally, if physical wash is conducted in the state that the hollow fiber membranes are clinging to each other in a rod shape, the hollow fiber membranes may be broken by an excessive external force toward the horizontal direction. The present invention also solves such problems.

Brief Summary Text (21):

1) A method for purifying aqueous suspension comprising feeding aqueous suspension containing a fine particle comprising an inorganic component from the outer surface of a wavy hollow fiber membrane having an outer diameter of from 0.5 to 3.1 mm to filter, followed by physical wash of the hollow fiber membrane.

Brief Summary Text (22):

2) A hollow fiber membrane bundle which is prepared by collecting a plurality of wavy hollow fiber membranes so as to orient in the same direction with a bulkiness of from 1.45 to 2.00, wherein the membrane has an inner diameter of from 0.3 to 1.7 mm, an outer diameter of from 0.5 to 3.1 mm, a membrane thickness of from 0.1 to 0.7 mm, and a flatness of from 0.8 to 1.0.

Brief Summary Text (23):

3) A method for producing a hollow fiber membrane bundle comprising the steps of: i) extruding membrane production raw liquid in the form of a hollow fiber through a co-axial tube-in-orifice spinning nozzle to obtain a hollow fiber material, ii) cooling and solidifying or coagulating the hollow fiber material to obtain a hollow fiber membrane, and iii) collecting a plurality of the thus-obtained hollow fiber membranes so as to orient in the same direction;

Brief Summary Text (24):

wherein a pulsation flow is contacted with the hollow fiber material before or during the cooling and solidifying step or the coagulating step.

Brief Summary Text (25):

4) A hollow fiber membrane module, wherein a plurality of wavy hollow fiber membranes each having an inner diameter of 0.3 to 1.7 mm, an outer diameter of 0.5 to 3.1 mm, a membrane thickness of 0.1 to 0.7 mm and a flatness of 0.8 to 1.0 is collected so as to orient in the same direction and mounted with a packing ratio of from 35 to 55%.

Drawing Description Text (4):

FIG. 3 is a schematic view of an example of a tool used for measuring bulkiness of a hollow fiber membrane bundle of the present invention.

Drawing Description Text (5):

FIG. 4 is a schematic view of an example of the method for producing a hollow fiber membrane of the present invention.

Drawing Description Text (6):

FIG. 5 is a schematic view of an example of the hollow fiber membrane module of the present invention.

Detailed Description Text (4):

The purifying method of the present invention is a method for purifying aqueous suspension comprising feeding aqueous suspension containing a fine particle comprising an inorganic component from the outer surface of a wavy hollow fiber membrane having an outer diameter of from 0.5 to 3.1 mm to filter, followed by physical wash of the hollow fiber membrane.

Detailed Description Text (5):

The filtration type can be either a dead-end type filtration wherein the whole quantity of raw water supplied is recovered as filtrate, or a cross flow type filtration wherein a part of rawwater supplied is recovered as concentrated water outside the membrane module system. Also, it may be either a pressurizing filtration type wherein raw water is pressurized from the outer surface side of the membrane by using a pump or the like to obtain filtrate, or a decompressing filtration type wherein a membrane module is submerged in a raw water tank or a raw water pit and the inner surface side of membrane is decompressed to obtain filtrate. The pressurizing filtration type is preferred because a higher filtration flux can be obtained.

Detailed Description Text (6):

An example of filtration is shown in FIG. 1. In FIG. 1, raw water (aqueous suspension) (1) is fed under pressure into a hollow fiber membrane module (4) through a circulation tank (2) with a raw water feed pump (3). The fine particles in raw water are trapped on the outer surface of hollow fiber membranes and the resultant filtrate is introduced into a filtrate tank (5) and stored therein

Detailed Description Text (7):

The raw water is fed from the outer surface side of a wavy hollow fiber membrane

having an outer diameter of from 0.5 to 3.1 mm. Although the outer diameter of the hollow fiber membrane can be changed depending on the effective length of a membrane module, the expected quantity of filtrate, or the like, is required to be within the range of from 0.5 to 3.1 mm in view of the pressure loss of filtrate in the hollow part of the membrane or the like. The outer diameter is preferably within the range of from 0.7 to 2.5 mm, more preferably within the range of from 1.0 to 2.5 mm.

Detailed Description Text (8):

In the present invention, wavy hollow fiber membranes are collected in the longitudinal direction so as to be bulky and mounted in a module. Therefore, the hollow fiber membranes contact almost at points and hardly rub against each other through suspended solids so that open pores on the membrane surface are not easily covered. As a result, a stable filtration operation is achieved. For the same reasons, suspended solids hardly accumulate in a hollow fiber membrane bundle, and even if the solids accumulate, they are easy to discharge by taking a step of physical wash such as back wash, air-scrubbing and flushing so that a stable filtration operation over a long period can be achieved.

Detailed Description Text (10):

The purifying method of the present invention is effective especially in the situation where a suspended solid accumulating on the surface of membranes during the filtration has a large diameter and the accumulation quantity of the solids is large. This is because, in such a situation, the membrane surface is most seriously damaged by the solids upon physical wash.

Detailed Description Text (11):

The amount of water to be filtered and the filtration time are appropriately adjusted according to the turbidity of raw water (aqueous suspension). As the turbidity of raw water becomes higher, it is necessary to reduce the amount of water to be filtered or shorten the time until the physical wash. Further, as the amount of raw water becomes larger, it is necessary to shorten the time until the physical wash. In Particular, for the purpose of preventing the suspended solids accumulating among the hollow fiber membranes from hardening and adhering to each other, it is preferred to select a filtration time so as for the amount of the suspended solids accumulating, which is defined by the following formula, to be in the range of from 0.0005 to 10, moreover in the range of from 0.01 to 10. The amount of suspended solids accumulating is a parameter for the amount of suspended solids accumulating on the unit membrane surface during a filtration step and is defined by the following formula:

Detailed Description Text (14):

The back wash is an operation comprising feeding a part of filtrate and/or a compressed gas from the filtrate side of a hollow fiber membrane (the inner surface side in case of the external pressure type filtration) to the raw water side (the outer surface side in case of the external pressure type filtration) to generate a flow of liquid and/or gas in the reverse direction to the ordinary filtration flow. For example, in FIG. 1, washing (back wash) is performed by feeding the filtrate in a filtrate tank (5) into a hollow fiber membrane module (4) with a back wash pump (6).

Detailed Description Text (16):

In view of the balance of a recovery ratio of filtrate and membrane recoverability by physical wash, water and/or compressed gas for back wash flows preferably in a flow amount [m.sup.3 /Hr] of from 0.5 to 5 times, particularly preferably in a flow amount [m.sup.3 /Hr] of from 1 to 3 times, as large as the flow amount [m.sup.3 /Hr] of filtrate during the filtration step.

Detailed Description Text (17):

The air-scrubbing step is an operation comprising feeding raw water containing

compressed gas such as compressed air and/or only compressed gas from the downside of a hollow fiber membrane module between the filtration steps to discharge the suspended solids accumulating among the hollow fiber membranes from the module. For example, in FIG. 1, air-scrubbing is performed by feeding compressed air generated in a compressor (7) into a raw water inlet of a hollow fiber membrane module (4). When the air-scrubbing step is carried out alone between the filtration steps using a conventional hollow fiber membrane module, the membrane surface may be damaged and the open pores on the surface may be covered if the amount of suspended solid accumulating per unit membrane area is large at the time of conducting air-scrubbing. According to the present invention, however, the treated water having high quality can be stably obtained at a high flow velocity of the membrane filtration even if severe air-scrubbing as described above is performed alone.

Detailed Description Text (19):

The flow amount [Nm.sup.3 /Hr] of gas fed in the normal state during the air-scrubbing step is preferably from 0.5 to 20 times, more preferably from 1 to 10 times, as large as the flow amount [m.sup.3 /Hr] of filtrate during the filtration step. The effect of air-scrubbing may be deteriorated when the flow amount is under the lower limit, and the hollow fiber membranes may be dried when the flow amount is over the upper limit.

Detailed Description Text (20):

The flushing step is an operation comprising widely opening a valve on the condensed water side and/or an air exhausting valve and feeding raw water in an amount larger than that in the filtration step to discharge the suspended solids accumulating among the hollow fiber membranes from the module. In this step, the valve on the filtrate side may be closed or throttled. Respective times necessary for a filtration step and a flushing step are appropriately selected according to the quality of raw water, the expected amount of filtrate or the like. It is preferred that the time of the flushing step is from 1/10000 to 1/5 of that of the filtration step. When the time of the flushing step is shorter than 1/10000 of that of the filtration step, the effect of flushing may deteriorate. When the time of the flushing step is longer than 1/5 of that of the filtration step, the proportion of the flushing step time to the total operation time becomes large. As a result, the amount of filtrate recovered per unit time is decreased.

Detailed Description Text (21):

In view of the balance of a recovery ratio of filtrate and membrane recoverability by physical wash, a flushing amount [m.sup.3 /Hr] of water during the flushing step is preferably from 1.1 to 8.0 times, more preferably from 1.5 to 5.0 times, as large as the flow amount [m.sup.3 /Hr] of filtrate during the filtration step.

Detailed Description Text (22):

The above-mentioned physical wash may be performed alone or in combination. Air-scrubbing simultaneous with back wash enables a stabler and longer-term filtration operation because it releases the compaction of suspended solids accumulating on the membrane surface and makes the solids float to discharge by air-scrubbing. It is also acceptable to perform back wash alone prior to air-scrubbing or air-scrubbing simultaneous with back wash. In this case, the release of the compaction of suspended solids accumulating on the membrane surface is advantageously accelerated. It is also acceptable to perform back wash alone after air-scrubbing or air-scrubbing simultaneous with back wash. In this case, the discharge of suspended solids in a membrane module is advantageously accelerated. Further, the physical wash method, in which flushing is performed after back wash and air-scrubbing are simultaneously performed, can be one of the effective physical wash methods because the recovery ratio of filtrate is improved by subjecting a part of suspended solids discharged by back wash and air-scrubbing to flushing.

Detailed Description Text (23):

The purifying method of the present invention can employ a step of dosing ozone or

the like in addition to the above-mentioned filtration step and physical wash step. One example of such a case is shown in FIG. 2. As shown in FIG. 2, raw water (11) is introduced into a circulation tank (12), fed into a hollow fiber membrane module (14) under pressure by using a raw water supplying pump (13), filtered in the module, and then stored in a filtrate tank (15). At this time, the raw water being fed into the module (14) under pressure is mixed with ozone gas generated by an ozone generator (18). The concentration of ozone water is controlled to be a certain concentration, for example 0.3 mg/l, on the filtrate side. At the time of back wash, the filtrate in the filtrate tank (15) is transferred to the module (14) by a back wash pump (16). At this time, air-scrubbing with compressed air generated by a compressor (17) may be performed.

Detailed Description Text (24):

<Hollow Fiber Membrane Bundle>

Detailed Description Text (25):

The hollow fiber membrane bundle used in the above-mentioned purification method is preferably a bundle which is prepared by collecting a plurality of wavy hollow fiber membranes so as to orient in the same direction with a bulkiness of from 1.45 to 2.00, wherein the membrane has an inner diameter of from 0.3 to 1.7 mm, an outer diameter of from 0.5 to 3.1 mm, a thickness of from 0.1 to 0.7 mm, and a flatness of from 0.8 to 1.0.

Detailed Description Text (26):

The material for a hollow fiber membrane includes polyolefin such as polyethylene, polypropylene, polybutene and the like; fluoro type resin such as a tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer (PFA), a tetrafluoroethylene-hexafluoropropylene copolymer (FEP), a tetrafluoroethylene-hexafluoropropylene-perfluoroalkylvinylether copolymer (EPE), an ethylene-tetrafluoroethylene copolymer (ETFE), polychlorotrifluoroethylene (PCTFE), chlorotrifluoroethylene-ethylene copolymer (ECTFE), polyvinylidene fluoride (PVDF) and the like; super-engineering plastics such as polysulfone, polyether sulfone, polyether ketone, polyether ether ketone, polyphenylene sulfide and the like; cellulose such as cellulose acetate, ethyl cellulose and the like; polyacrylonitrile, polyvinyl alcohol and compositions thereof.

Detailed Description Text (27):

As the hollow fiber membrane, a membrane having a pore diameter in the range of a nanofilter, an ultrafiltration (UF) membrane and a microfiltration (MF) membrane can be used. Among these, the ultrafiltration (UF) membrane and the microfiltration (MF) membrane which basically contribute a high filtrate amount are preferred. In particular, the MF membrane is more preferred. For example, the preferred membrane has an average pore diameter of from 0.001 to 1 μm , and the more preferred membrane has that of from 0.05 to 1 μm . Herein, the average pore diameter is measured by an air flow method (ASTM: F316-86). Further, a hollow fiber membrane having a porosity of from 50 to 90% is preferred. Herein, the porosity is calculated from a weight of a hollow fiber membrane saturated with water, a simple volume of a hollow fiber membrane (a volume calculated from the inner diameter, outer diameter and length thereof) and a specific gravity of the polymer used.

Detailed Description Text (28):

The hollow fiber membrane of the present invention is wavy. The wavy hollow fiber membrane means a membrane which meanders when being left without applying any tension.

Detailed Description Text (29):

From the viewpoint of improving the bulkiness of a hollow fiber membrane bundle, it is preferred that the waves having different wavelength and/or wave height exist together.

Detailed Description Text (30):

The hollow fiber membrane bundle has a bulkiness of preferably from 1.45 to 2.00, more preferably from 1.55 to 2.00.

Detailed Description Text (31):

The bulkiness of the hollow fiber membrane mentioned above is defined by the following formula.

Detailed Description Text (32):

In the above formula (I), S1 represents cross-section area of a small bundle of six hundred hollow fiber membranes, which are selected randomly from hollow fiber membranes constituting a membrane bundle, under load of 2.9 N (300 gf). S1 can be calculated from the circumferential length of the small bundle measured under load of 2.9 N (300 gf) using a polyethyleneterephthalate (PET) film, which has a thickness of 100 .mu.m and width of 40 mm, and is equipped with a spring scale at the edge. S2 represents a value which is obtained by multiplying the cross-sectional area of a hollow fiber membrane calculated using the outer diameter thereof by six hundred.

Detailed Description Text (33):

Further, the circumferential length of a hollow fiber membrane bundle could be more easily measured by using a tool for the bulkiness measurement prepared by jointing two PET films through a rectangular frame in advance. A specific example of the measuring methods is explained referring to FIG. 3.

Detailed Description Text (35):

ii) Into the cylinder, a hollow fiber membrane bundle (24) is installed. After fixing one of the two PET films, 2.9 N (300 gf) weight was applied to the other PET film with a hole (23), which is arranged in advance for holding the hook of a spring scale, through a spring scale to mark the PET film.

Detailed Description Text (37):

When the bulkiness is less than 1.45, the uniformity of a filling adhesive agent at the time of preparing a module and the efficiency of discharge of suspended solids by physical wash after a module is made can be insufficient. When the bulkiness is more than 2.00, a maximum number of hollow fiber membranes to be installed in a module can decrease so that it can be difficult to obtain a sufficient membrane area per module.

Detailed Description Text (38):

The outer diameter of the hollow fiber membrane is within the range of from 0.5 to 3.1 mm, preferably within the range of from 0.7 to 2.5 mm, more preferably within the range of from 1.0 to 2.5 mm, from the viewpoint of the amount of filtrate to be recovered, the efficiency of compressive creep, the necessary bursting pressure or the like. In addition, the inner diameter of the hollow fiber membrane is preferably within the range of from 0.3 to 1.7 mm. In case of the external pressure filtration, since filtrate flows in the hollow portion of the hollow fiber membranes, the inner diameter of less than 0.3 mm causes the increase of pressure loss of filtrate in the hollow portion so that a filtrate amount to be recovered can be reduced with the decrease of the effective membrane area in a module. On the other hand, when the inner diameter is more than 1.7 mm, it is difficult to obtain a large membrane area per module so that a filtrate amount to be recovered can be decreased as well. The thickness of the hollow fiber membrane is preferably within the range of from 0.1 to 0.7 mm, more preferably within the range of from 0.2 to 0.5 mm, from the viewpoint of the balance of the compressive creep efficiency and necessary bursting pressure, which are exhibited when the inner diameter of the membrane is within the above-mentioned range.

Detailed Description Text (39):

The flatness of the hollow fiber membrane is preferably from 0.8 to 1.0. Herein,

the flatness means a ratio of the inner minor axis to the inner major axis, especially the ratio at the curved portion of a wavy membrane, when the cross-section of the hollow fiber membrane is oval. The ratio is defined by the following formula.

Detailed Description Text (41):

When the flatness is less than 0.8, the burst strength or the compressive strength can be greatly decreased; and the pressure loss of the expansion and the reduction can be repeated when the filtrate passes through the waved portions of the hollow fiber membrane. As a result, the operation pressure at the time of filtration operation can be increased and the stability of filtration can be defective. The flatness is preferably not less than 0.9, more preferably not less than 0.95, to improve the bursting strength and the compressive strength and suppress the rise of pressure loss.

Detailed Description Text (42):

It is preferred that the hollow fiber membrane bundle of the present invention consists of wavy hollow fiber membranes in order to reduce the contact portion where the hollow fiber membranes in contact each other and to prevent the covering of open pores on the membrane surface caused by scrubbing of the membrane surfaces.

Detailed Description Text (43):

<Method for Producing Hollow Fiber Membrane Bundle>

Detailed Description Text (44):

The hollow fiber membrane bundle of the present invention can be preferably obtained by a method for producing a hollow fiber membrane bundle comprising the steps of extruding membrane production raw liquid in the form of a hollow fiber through a co-axial tube-in-orifice spinning nozzle to obtain a hollow fiber material, cooling and solidifying or coagulating the hollow fiber material to obtain a hollow fiber membrane, and collecting a plurality of the thus-obtained hollow fiber membranes so as to orient in the same direction; wherein a pulsation flow is contacted with the hollow fiber material before or during the cooling and solidifying step or the coagulating step.

Detailed Description Text (46):

One of the features of the present invention is to contribute waves to a membrane by contacting a hollow fiber material (32) extruded from the spinning nozzle (31) to flow down in a half-solidified state with a pulsation flow injected from a pulsation flow exhalation nozzle (33). Namely, by making a pulsation flow contacted with a half-solidified hollow fiber material, the hollow fiber material is vibrated. In a curved state, the material is cooled and solidified or coagulated in a cooling and solidifying bath or a coagulation bath. As a result, waves can be formed easily

Detailed Description Text (47):

The wavy hollow fiber membrane can be also obtained by other methods than above, for example, heat treatment of the hollow fiber membrane or the like. In case of a hollow fiber membrane having a large diameter, however, the hollow portion of the membrane can be unpreferably crushed or flattened in excess when it is tried to form waves according to the above-mentioned heat treatment. On the contrary, the production method of the present invention employing the above-mentioned pulsation flow can contribute waves without causing any flatness of inner/outer diameters even if the hollow fiber membrane has a large diameter, for example, an outer diameter of even 2.5 mm. In addition, preferred waves having different wavelength and wave height can be easily formed. Further, it is possible to prevent a damage of the membrane surface because solids do not contact with the membrane surface. According to the production method of the present invention employing a pulsation flow, the flatness of the hollow fiber membrane obtained can be from 0.8 to 1.0, in

most cases be from 0.9 to 1.0.

Detailed Description Text (49):

The recurrence interval of a pulsation flow (exhalation interval) is properly adjusted according to a winding speed of a hollow fiber membrane in the membrane production. For instance, when the winding speed is within the range of from 10 to 30 m/min, the recurrence interval is preferably within the range of from 0.05 to 1.5 sec/shot. When the interval is shorter than 0.05 sec/shot, the pulsation flow does not occur with the result that waves are not formed. When the interval is longer than 1.5 sec/shot, the resultant waves have a long wavelength with the result that the bulkiness is insufficient.

Detailed Description Text (50):

The temperature of a pulsation flow is not especially limited. This temperature can be the temperature of a cooling and solidifying bath, for example, within the range of from 20 to 80.degree. C. in the case of the membrane production process by melt extrusion, and a temperature of a coagulating bath, for example, within the range of from -10 to 80.degree. C. in the case of the wet membrane production.

Detailed Description Text (51):

The waves can be formed by contacting a half-solidified hollow fiber material with a pulsation flow to shake, and cooling and solidifying or coagulating the material in the state of shaking. It is supposed that one contact of a pulsation flow with the hollow fiber material forms not merely one wave, but 2 to 10 waves. The half-solidified hollow fiber material meanders by the contact with a pulsation flow and the meandering attenuates gradually. Therefore, the resultant hollow fiber membrane does not have one kind of the wavelengths and/or wave heights, but various kinds of wavelengths and/or wave heights together.

Detailed Description Text (52):

When the pulsation flow contacts the half-solidified material during the cooling and solidifying or coagulating step, that is, in a cooling and solidifying bath or a coagulating bath, the contact position is preferably, for example, from the bath surface to not deeper than 500 mm below the bath surface. When the pulsation flow contacts the half-solidified material before the cooling and solidifying step or the coagulating step, that is, above the bath surface of the cooling and solidifying bath or the coagulating bath, the contact position is preferably, for example, from the bath surface to not higher than 50 mm above the bath surface. When the contact position is far above the cooling and solidifying bath or the coagulating bath, i.e., near the spinning nozzle, only the portion where the pulsation flow contacts is promptly cooled and solidified or coagulated so that it is possible that pores may not be uniformly formed on the circumference of the hollow fiber membrane and, in an extreme case, no pores may be formed at the contact portion or the skin layer may become thick only at the contact portion. On the contrary, when the contact position is deep in the bath, e.g., deeper than 500 mm below the bath surface, the hollow fiber material is cooled and solidified or coagulated before the contact with the pulsation flow, and waves are not formed.

Detailed Description Text (53):

If a guide such as a thread guide is arranged in the cooling and solidifying bath or the coagulating bath so as for the hollow fiber material not to run off by the contact with the pulsation flow at the time when the pulsation flow is contacted with the hollow fiber material, more preferred waves can be formed.

Detailed Description Text (54):

<Hollow Fiber Membrane Module>

Detailed Description Text (55):

In the purifying method of the present invention, it is preferred to use a hollow fiber membrane module in which a plurality of wavy hollow fiber membranes each

having an inner diameter of 0.3 to 1.7 mm, an outer diameter of 0.5 to 3.1 mm, a thickness of 0.1 to 0.7 mm and a flatness of 0.8 to 1.0 is collected so as to orient in the longitudinal direction and mounted with a packing ratio of from 35 to 55%.

Detailed Description Text (56):

The packing ratio means a ratio at which the inner wall sectional area of a module case is packed with hollow fiber membranes based on the outer diameter of the membrane, which can be calculated by the following formula.

Detailed Description Text (57):

By installing a hollow fiber membrane bundle having high bulkiness at a packing ratio of from 35 to 55%, the contact of the hollow fiber membranes remains only at points since the bundle therein is bulky. As a result, the hollow fiber membranes hardly rub against each other through suspended solids and open pores on the outer surface of the hollow fiber membrane are not easily covered. For the same reasons, the suspended solids hardly accumulate in the hollow fiber membrane bundle and are easily discharged by physical wash such as back wash, air-scrubbing, flushing or the like, even if accumulate. As a result, a filtration operation can be stably conducted for a long period. Further, since the distribution situation of the hollow fiber membranes in the inner section of the membrane module improves owing to the waves, the defective portion hardly occurs in the bonded and fixed portion of the module even in the case that the pre-hardening initial viscosity of the bonding agent is high or the module is a large-scale module having a large diameter when the hollow fiber bundle is bonded and fixed to a module case.

Detailed Description Text (58):

Although the packing ratio of less than 35% provides excellent efficiency of discharge by wash, the effect of using a hollow fiber membrane module is reduced since a large membrane area per unit volume of the hollow fiber module is not secured. The packing ratio of more than 55% can secure a large membrane area per unit volume of the hollow fiber module, but the hollow fiber membranes aggregate densely in the module so that the suspended solids are hard to discharge in case of the external pressure filtration.

Detailed Description Text (59):

In the hollow fiber membrane module of the present invention, at least one edge of the hollow fiber membrane bundle is fixed with a thermosetting resin like an epoxy resin. The hollow fiber membrane module of the present invention is mounted with hollow fiber membranes with a hollow portion open and has a structure enabling a filtration from the outer to inner surface of the hollow fiber membrane; therefore, it is suitable for an external pressure filtration. The hollow fiber membrane module may be bonded and fixed at both edges or either edge. It is also allowed to seal the hollow portion of hollow fiber membranes at one of the bonded and fixed edges. It is also possible to use a membrane module in which both edges are bonded and fixed, the hollow portions of the hollow fiber membranes are sealed at one edge and an inlet for raw water is opened as described in Japanese Patent Application Laid-Open No. 7-171354.

Detailed Description Text (60):

The hollow fiber membrane module of the present invention includes a cartridge type module which is used after being installed and arranged in a tank with tube sheets or an outline housing beside a directly connected rack type, which is connected to a rack through pipes or the like. The above-mentioned cartridge type module indicates, differing from the common directly connected rack type module, such a module that maintains the shape of a hollow fiber membrane bundle portion with a cylinder provided with holes by punching or the like, a net and the like, and does not take fluid-tight treatments except for the bonded and fixed portion. In this case, the hollow fiber membrane bundle is allowed to be naked except for the bonded and fixed portion if the bundle can maintain its shape by itself.

Detailed Description Text (61):

Since the hollow fiber membrane module of the present invention employs the wavy hollow fiber membranes as described above, the bonded and fixed portion at the edge of the hollow fiber membranes is less defective even in the case of a large-scale module having an outer diameter of from 170 to 350 mm.

Detailed Description Text (62):

The thermosetting resin used to bond and fix one or both edges of the hollow fiber membrane module of the present invention includes an epoxy resin, a urethane resin, a silicone rubber and the like. If necessary, there may make attempts to improve the strength of a resin partition and reduce shrinkage on curing by adding a filler such as silica, carbon black and fluorocarbon to these resins.

Detailed Description Text (63):

The material of the hollow fiber membrane module case includes polyolefins such as polyethylene, polypropylene and polybutene; fluoro resins such as polytetrafluoroethylene (PTFE), PFA, FEP, EPE, ETFE, PCTFE, ECTFE, PVDF and the like; chloro resins such as polyvinyl chloride and polyvinylidene chloride; a polysulfone resin, a polyethersulfone resin, a polyallyl sulfone resin, a polyphenyl ether resin, an acrylonitrile butadiene styrene copolymer resin (ABS resin), an acrylonitrile styrene copolymer resin, a polyphenylene sulfide resin, a polyamide resin, a polycarbonate resin, a polyether ketone resin, a polyether ether ketone resin, compounds thereof, and metals such as aluminum and stainless steels. In addition, compounds of resin and metals, resin reinforced with glass fiber or carbon fiber can be used.

Detailed Description Text (64):

The hollow fiber membrane module of the present invention can be prepared, for example, by bonding and fixing at least one edge of the hollow fiber bundle collected in the longitudinal direction with a thermosetting resin such as an epoxy resin and then cutting a part of the bonded and fixed portion so as to open the hollow portion of the hollow fiber membrane.

Detailed Description Text (65):

A plurality of openings is preferably arranged at one of the edge bonded and fixed portions of the hollow fiber membrane module to supply raw water and/or gas for air-scrubbing more uniformly. The above-mentioned opening preferably has an equivalent diameter of 3 to 100 mm. When the diameter of the opening is less than 3 mm, the opening can be clogged with suspended solids included in the raw water. When the diameter of the opening is more than 100 mm, it is required to reduce the number of hollow fiber membranes installed in the module and/or the number of the openings so that the raw water is hard to be supplied uniformly. The section configuration of the openings is not especially limited, and includes polygons such as a triangle, quadrangle, hexagon and the like in addition to circle and oval. Among them, circle and oval are preferred. Further, the openings can be arranged uniformly or at random at the edge bonded and fixed portions.

Detailed Description Text (66):

One of the examples of the hollow fiber membrane module of the present invention is shown in FIG. 5. In FIG. 5, a hollow fiber membrane module is connected with a pipe of an operation device through a cap (46). The raw water and/or compressed gas to be supplied pass through a raw water inlet (45) and are filtered from the outer to inner surface of the wavy hollow fiber membrane (41).

Detailed Description Text (67):

In this case, the pressure of the raw water pressured by a pump or the like is maintained by a module case (43), and a part of the raw water is recovered as filtrate. The condensed raw water is discharged from the hollow fiber membrane module through a condensed water outlet (47). The hollow fiber membranes with

hollow portion open are bonded and fixed fluid-tight to the module case at the bonded portion (42) so as not to mix raw water and filtrate. At a bonded portion (44), the hollow portions of the hollow fiber membranes are sealed and at the same time are equipped with a plurality of openings, and a raw water inlet (45) is arranged.

Detailed Description Text (68):

According to the purifying method of the present invention, it is possible to prevent damage of the membrane surface by fine particles at the time of treating the aqueous suspension containing fine particles comprising an inorganic material with the membrane and to stably perform a filtration over a long term. Accordingly, the present invention is suitable for the field of purifying the aqueous suspension containing an inorganic material, e.g., tap water such as river water, lake water, marsh water and groundwater; water for industrial uses; waste water; secondary treatment waste water; industrial sewage; domestic sewage; human waste; sea water and the like. In addition, the hollow fiber membrane module of the present invention has an advantage of less scrubbing and damage to the membranes due to bulky waved hollow fiber membranes having a large diameter. Therefore, it can be suitably used for the purifying method of the present invention. The module also has excellent discharge efficiency of suspended solids. Further, the hollow fiber membrane of the present invention is suitable for a large-scale module which has fewer defects in the bonded portion at the edge of membrane.

Detailed Description Text (69):

Hereinafter, examples of production of the hollow fiber membrane, the hollow fiber membrane module and the aqueous suspension purifying method, which are employed in the present invention, are described. In the Examples and Comparative Examples, turbidity and particle size were measured by the following method.

Detailed Description Text (70):

Flatness of Hollow Fiber Membrane: A curved portion of the wave of the hollow fiber membrane was cut out at five points to measure the minor axis and major axis of the inner diameter thereof using an X-Y microscope [STM-222DH (trade name) manufactured and sold by Olympus Optical Company Limited], and flatness (minor axis/major axis) of each portion was calculated.

Detailed Description Text (73):

Water Flux Amount of Single Hollow Fiber Membrane: Pure water at 25.degree. C. is permeated from the inner to outer surface side of a porous hollow fiber membrane sample having an effective length of 100 mm to calculate a flux amount per unit time and that per unit pressure (differential pressure per unit membrane).

Detailed Description Text (74):

Wash Recoverability: Evaluated based on a ratio (%) of a pure water flux amount of a module after subjected to evaluation by real liquid such as river water and chemical wash to a pure water flux amount (initial value) of a module before subjected to evaluation by real Liquid; or a ratio (%) of a pure water flux amount of a single membrane fiber, which is obtained by dismantling a module after evaluation by real liquid and washing only the membrane with chemicals, to a pure water flux amount (a flux amount of an unused membrane) of a single hollow membrane fiber before preparing a module.

Detailed Description Text (76):

Production of Hollow Fiber Membrane

Detailed Description Text (79):

At the time of the extrusion, a pulsation flow exhalation nozzle was arranged at the position of 10 mm above the bath surface, and a cooling and solidifying liquid was contacted with the hollow fiber material flowing down at an exhalation interval of 0.3 sec/shot using a diaphragm pump [NDP-5FST manufactured and sold by Yamada

Corporation] to obtain a wavy hollow fiber membrane.

Detailed Description Text (80):

The above-mentioned wavy hollow fiber membrane was wound up through a three-ream roller at a winding speed of 20 m/min. The obtained hollow fiber membrane bundle was treated with dichloromethane under the following conditions to extract DOP and DBP from the hollow fiber membrane.

Detailed Description Text (81):

Extraction Conditions: Treatment Temperature: room temperature (25 to 27.degree. C.) Volume of dichloromethane relative to simple volume of hollow fiber membrane (calculated from inner diameter, outer diameter and length thereof): 20 fold Treatment Period: 5 hours

Detailed Description Text (82):

Then, the obtained hollow fiber membrane bundle was soaked in a 50% ethanol solution for 30 minutes and treated with a sodium hydroxide solution having a weight percent concentration of 20% under the following conditions to extract silica from the hollow fiber membrane.

Detailed Description Text (83):

Extraction Conditions: Temperature: 60.degree. C. Volume of sodium hydroxide solution relative to simple volume of hollow fiber membrane (calculated from inner diameter, outer diameter and length thereof): 20 fold (8 fold equivalent in equivalent ratio relative to hydrophobic silica) Treatment Period: 2 hours

Detailed Description Text (84):

The above treated hollow fiber membrane bundle was rinsed for an hour with 60.degree. C. hot water having the same volume as the above-mentioned sodium hydroxide solution. The above wash with hot water was repeated a total of ten times to obtain a porous hollow fiber membrane bundle. The thus-obtained hollow fiber membrane had an inner diameter/outer diameter of 0.70 mm.phi./1.25 mm.phi., a porosity of 70%, an average pore diameter of 0.18 .mu.m, a pure water flux amount of 2,000 [l/m.sup.2.multidot.min.multidot.100 kPa.multidot.25.degree. C.], and flatness as shown in Table 1. The circumferential length and the bulkiness of the hollow fiber membrane bundle were 124.0 mm and 1.66, respectively. In addition, waves with different wavelength and wave height coexisted in the hollow fiber membrane.

Detailed Description Text (86):

Production of Hollow Fiber Membrane

Detailed Description Text (87):

A hollow fiber membrane bundle was prepared in substantially the same manner as described in Example 1 except that the pulsation flow was not contacted with the hollow fiber. The thus-obtained hollow fiber membrane had an inner diameter/outer diameter of 0.70 mm.phi./1.25 mm.phi., a porosity of 70%, an average pore diameter of 0.18 .mu.m, a pure water flux amount of 2,000 [l/m.sup.2.multidot.min.multidot.100 kPa.multidot.25.degree. C.] and flatness as shown in Table 1. In addition, the circumferential length and the bulkiness of the hollow fiber membrane bundle were 115.0 mm and 1.43, respectively.

Detailed Description Text (89):

Production of Hollow Fiber Membrane

Detailed Description Text (90):

The hollow fiber membrane obtained in Example 2 was passed between two gears at the atmosphere temperature of 140.degree. C. to obtain a wavy hollow fiber membrane bundle. The gears used had a curvature minimum radius at the edge of 5 mm and an edge distance of 25 mm. Two of such gears were bitten each other so as to be a

biting height and minimum gear distance of 15 mm and 3 mm, respectively. The thus-obtained hollow fiber membrane had an inner diameter/outer diameter of 0.70 mm.phi./1.25 mm.phi., a porosity of 70% and an average pore diameter of 0.18 .mu.m. Its pure water flux amount was slightly reduced to 1,950 [1/m.sup.2.multidot.min.multidot.100 kPa.multidot.25.degree. C.], which was supposed to be caused by an influence of the flatness of the hollow fiber membrane as shown in Table 1. Further, the circumferential length and bulkiness of the hollow fiber membrane bundle were 118.2 mm and 1.51, respectively. In addition, waves of the hollow fiber membrane had approximately the same wavelength and wave height.

Detailed Description Text (92):

Production of Hollow Fiber Membrane

Detailed Description Text (93):

The hollow fiber membrane obtained in Example 2 was passed between two gears at the atmosphere temperature of 140.degree. C. to obtain a wavy hollow fiber membrane bundle. The gears used had a curvature minimum radius at the edge of 7 mm and an edge distance of 30 mm. Two of such gears were bitten each other so as to be a biting height and minimum gear distance of 10 mm and 8 mm, respectively. The thus-obtained hollow fiber membrane had an inner diameter/outer diameter of 0.70 mm.phi./1.25 mm.phi., a porosity of 70% and an average pore diameter of 0.18 .mu.m. Its pure water flux amount was 2,000 [1/m.sup.2.multidot.min.multidot.100 kPa.multidot.25.degree. C.], and the flatness of the hollow fiber membrane as shown in Table 1. Further, the circumferential length and bulkiness of the hollow fiber membrane bundle were 115.5 mm and 1.44, respectively. In addition, waves of the hollow fiber membrane had approximately the same wavelength and wave height.

Detailed Description Text (95):

Production of Hollow Fiber Membrane Module

Detailed Description Text (96):

300 of the hollow fiber membranes obtained in Example 1 were bundled.

Detailed Description Text (97):

Then, the hollow portion of one of the edge faces of the thus-obtained bundle was sealed and the bundle was mounted in a cylindrical polysulfone module case having an inner diameter of 36 mm.phi. and a length of 1,000 mm. On the sealed edge of the hollow fiber membrane, only a bonding jig was attached fluid-tight to the module case. On the other edge of the hollow fiber membrane, a total of five polypropylene rods, each having an outer diameter of 5 mm.phi., were arranged in parallel to the hollow fiber membrane and then a bonding jig was attached fluid-tight to the module case. In this case, the packing ratio was calculated at 36% from the outer diameter of the hollow fiber membrane, the packing number of the hollow fiber membranes and the inner diameter of the module case.

Detailed Description Text (98):

The above-mentioned module case equipped fluid-tight with bonding jigs at both edges was centrifugally molded using a two-liquid type epoxy bonding agent. After the centrifugal molding, the bonding jigs and the polypropylene rods were removed, and the bonded portion at the sealed edge was cut to open the hollow portion of the hollow fibers. As described above, a module comprising a bundle of wavy hollow fiber membranes was obtained.

Detailed Description Text (102):

Production of Hollow Fiber Membrane Module

Detailed Description Text (103):

1,800 of the hollow fiber membranes obtained in Example 1 were bundled.

Detailed Description Text (104):

Then, the hollow portion of one of the edge faces of the thus-obtained bundle was sealed, and the bundle was mounted in a cylindrical polyvinyl chloride module case having an inner diameter of 83 mm.phi. and a length of 1,000 mm. On the sealed edge of the hollow fiber membrane, only a bonding jig was attached fluid-tight to the module case. On the other edge of the hollow fiber membrane, a total of five polypropylene rods, each having an outer diameter of 11 mm.phi., were arranged in parallel to the hollow fiber membrane and then a bonding jig was attached fluid-tight to the module case. In this case, the packing ratio was calculated at 41% from the outer diameter of the hollow fiber membrane, the packing number of the hollow fiber membranes and the inner diameter of the module case.

Detailed Description Text (105):

The above-mentioned module case equipped fluid-tight with bonding jigs at both edges was centrifugally molded using a two-liquid type epoxy bonding agent. After the centrifugal molding, the bonding jigs and the polypropylene rods were removed, and the bonded portion at the sealed edge was cut to open the hollow portion of the hollow fibers. As described above, a module comprising a bundle of wavy hollow fiber membranes was obtained.

Detailed Description Text (109):

Production of Hollow Fiber Membrane Module

Detailed Description Text (110):

Four bundles each comprising 1,440 of the porous hollow fiber membranes obtained in Example 1 were prepared.

Detailed Description Text (111):

Then, after sealing the hollow portion of one of the edge faces of each bundle, the four bundles were mounted in a cylindrical SUS-304 module case having an inner diameter of 150 mm.phi. and a length of 1,500 mm. On the sealed edge of hollow fiber membrane, only a bonding jig was attached fluid-tight to the module case. On the other edge of the hollow fiber membrane, a total of 37 polypropylene rods, each having an outer diameter of 10 mm.phi., were arranged in parallel to the hollow fiber membrane and then a bonding jig was attached fluid-tight to the module case. In this case, the packing ratio was calculated at 40% from the outer diameter of the hollow fiber membrane, the packing number of the hollow fiber membranes and the inner diameter of the module case.

Detailed Description Text (113):

After the centrifugal molding, the bonding jigs and the polypropylene rods were removed, and then the bonded portion of the sealed edge was cut to open the hollow portions of the hollow fiber membranes after the silicone bonded portion was sufficiently cured. As a result, a hollow fiber membrane module comprising a bundle of wavy hollow fiber membranes was obtained.

Detailed Description Text (116):

Production of Hollow Fiber Membrane Module

Detailed Description Text (117):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 5 except that the hollow fiber membrane obtained in Example 2 was used. The packing ratio of the thus-obtained module was 36%.

Detailed Description Text (120):

Production of Hollow Fiber Membrane Module

Detailed Description Text (121):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 6 except that the hollow fiber membrane obtained in Example 2

was used. The packing ratio of the thus-obtained module was 41%.

Detailed Description Text (124):

Production of Hollow Fiber Membrane Module

Detailed Description Text (125):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 7 except that the hollow fiber membrane obtained in Example 2 was used. The packing ratio of the thus-obtained module was 40%.

Detailed Description Text (128):

Production of Hollow Fiber Membrane Module

Detailed Description Text (129):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 5 except that the hollow fiber membrane obtained in Example 3 was used. The packing ratio of the thus-obtained module was 36%.

Detailed Description Text (132):

Production of Hollow Fiber Membrane Module

Detailed Description Text (133):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 6 except that the hollow fiber membrane obtained in Example 3 was used. The packing ratio of the thus-obtained module was 41%.

Detailed Description Text (136):

Production of Hollow Fiber Membrane Module

Detailed Description Text (137):

A hollow fiber membrane module was prepared in substantially the same manner as described in Example 5 except that the hollow fiber membrane obtained in Example 4 was used. The packing ratio of the thus-obtained was 36%.

Detailed Description Text (141):

Using the hollow fiber membrane module obtained in Example 5, an operation was conducted. As raw water, a model liquid (a mixed solution of bentonite and humic acid [bentonite concentration: 1,000 mg/l, humic acid concentration: 500 mg/l in terms of a total organic carbon amount (TOC)] at the time of production) was used. As shown in FIG. 1, raw water (1) was fed under pressure into a hollow fiber membrane module (4) through a circulation tank (2) by using a raw water feed pump (3). The resultant filtrate was stored in a filtrate tank (5). At the time of back wash, the filtrate in the filtrate tank (5) was fed into the hollow fiber membrane module by using a back wash pump (6). In addition, air-scrubbing was conducted by supplying compressed air generated in a compressor (7) to a raw water inlet of the hollow fiber membrane module.

Detailed Description Text (142):

The filtration was conducted according to a cross-flow type filtration in which raw water (1) was fed into a hollow fiber membrane module (4) at a constant flow of 1.8 [l/min.multidot.module.multidot.25.degree. C.] so as to be a ratio of a membrane filtration flow to a water circulation flow of 1/1, and conducted according to an external pressure filtration with a constant filtration flow, i.e., a filtrate amount of 0.9 [l/min.multidot.module.multidot.25.degree. C.].

Detailed Description Text (143):

The operation was performed by repeating a 10 minute filtration followed by back wash with filtrate at a flow of 1.5 [l/min.multidot.module.multidot.25.degree. C.] for 20 seconds and conducting air-scrubbing with compressed air at a flow of 5 [Nl/min.multidot.module.multidot.25.degree. C.] for one minute every one hour. The

turbidity of raw water was 770, degrees. The total amount of the filtrate permeating the membrane during the filtration step was 9 liters. The amount of suspended solids accumulating was 5.87.

Detailed Description Text (147):

Further, the above-mentioned hollow fiber membrane module was washed with a sodium hypochlorite aqueous solution, a sodium hydroxide aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution until the recoverability was saturated. When the pure water flux amount was measured, it was 98% of the initial value.

Detailed Description Text (148):

Subsequently, when the hollow fiber membrane module was dismantled to observe the membrane outer surface of the hollow fiber membrane with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane surface was negligible.

Detailed Description Text (151):

Using the hollow fiber membrane module obtained in Example 5, an operation was conducted. As raw water, river surface water having an average turbidity among days of 1 and a fine particle diameter of from 5 to 200 μm (medium value: 50 μm) was used. The filtration was conducted using cross-flow type filtration in which raw water was fed into a hollow fiber membrane at a constant flow of 3.0 [l/min.multidot.module.multidot.25.degree. C.] so as to be a ratio of membrane filtration flow to a water circulation flow of 1/1, according to an external pressure filtration operation with a constant filtration flow, i.e., a filtrate amount of 1.5 [l/min.multidot.module.multidot.25.degree. C.].

Detailed Description Text (152):

The operation was performed by repeating a 20 minute filtration followed by back wash with filtration water at a flow of 2.5 [l/min.multidot.module.multidot.25.degree. C.] for 20 seconds and conducting air-scrubbing with compressed air at a flow of 7 [Nl/min.multidot.module.multidot.25.degree. C.] for one minute every hour. The turbidity of raw water was 1.0 degree. The total amount of the filtrate permeating the membrane during the filtration step was 30 liters. The amount of suspended solids accumulating was 0.025.

Detailed Description Text (155):

Further, the above-mentioned hollow fiber membrane module was washed with a sodium hypochlorite solution, a sodium hydroxide solution, an oxalic acid solution and a nitric acid solution until the recoverability was saturated. When the pure water flux amount was measured, it was 96% of the initial value.

Detailed Description Text (156):

Subsequently, when the hollow fiber membrane module was dismantled to observe the membrane outer surface of the hollow fiber membrane with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane surface was negligible.

Detailed Description Text (159):

Using the hollow fiber membrane module obtained in Example 6, a purifying operation was conducted. As raw water, river surface water having a turbidity of 0.1 to 5 degrees (average: 2.4 degrees), a fine particle diameter in water of from 0.9 to 30 μm (medium value: 9 μm) and a temperature of 12.degree. C. was used.

Detailed Description Text (160):

The filtration was conducted using cross-flow type filtration in which raw water was fed into a hollow fiber membrane at a constant flow of 2.6 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.] so as to a ratio of a membrane

filtration flow to a water circulation flow of 1/1, with a constant filtration flow, i.e., a filtrate amount of 1.3 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.].

Detailed Description Text (161):

The operation was performed by repeating a 20 minute filtration followed by back wash with filtrate for 20 seconds and conducting back wash with filtrate at a flow of 1.3 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.] and air-scrubbing with compressed air at a flow of 2 [Nm.sup.3 /hr.multidot.module.multidot.25.degree. C.] simultaneously for 2 minutes every hour. The turbidity of raw water was 2.4 degrees. The total amount of the filtrate permeating the membrane during the filtration step was 0.43 m.sup.3. The amount of suspended solids accumulating was 0.15.

Detailed Description Text (162):

After 12 month operation under the above-mentioned conditions, the trans-membrane pressure became 1.3 times the initial value. After the operation, the module was taken out from the device to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 95% of that of unused membrane. When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane outer surface was negligible.

Detailed Description Text (165):

Using the hollow fiber membrane module obtained in Example 6, an operation was conducted in substantially the same manner as Example 16 except that the operation was performed by conducting a membrane filtration for 60 minutes and conducting back wash with filtrate and air-scrubbing with compressed air simultaneously for 2 minutes. The turbidity of raw water was 2.4 degrees. The total amount of filtrate permeating the membrane during the filtration step was 1.3 m.sup.3. The amount of suspended solids accumulating was 0.44.

Detailed Description Text (166):

After 6 month operation under the above-mentioned conditions, the trans-membrane pressure became 1.4 times the initial value. After the operation, the module was taken out from the device to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled, and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 95% of that of unused membrane. When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane surface was negligible.

Detailed Description Text (169):

Using the membrane module obtained in Example 7, an operation was conducted. As raw water, river surface water having turbidity of from 1 to 3 degrees (average: 1.8 degrees) and a fine particle diameter of from 2 to 50 .mu.m (medium value: 22 .mu.m) was used. As shown in FIG. 2, raw water (11) was fed under pressure into a hollow fiber membrane module (14) through a circulation tank (12) by using a raw water feed pump (13). The resultant filtrate was stored in a filtrate tank (15). The raw water fed into the hollow fiber membrane module (14) under pressure was mixed with an ozone gas generated by an ozone generator (18) to obtain a concentration of ozone water of 0.3 mg/l at the filtrate side. At the time of back wash, the filtrate in the filtrate tank (15) was fed into the hollow fiber membrane

module (14) by using a back wash pump (16).

Detailed Description Text (170):

The filtration was conducted according to a dead-end type one under constant pressure, i.e., a trans-membrane pressure of 30 kPa, in which the raw water (11) was supplied to the hollow fiber membrane module (14) and concentrated water was not discharged except for discharge of ozone-containing air. At the time of back wash, a back wash pressure was 50 kPa.

Detailed Description Text (172):

Subsequently, when the hollow fiber membrane module was taken out from the filtration operation device and weighed, its weight had increased to 115% of the initial weight.

Detailed Description Text (173):

Again, this module was installed in the operation device shown in FIG. 2 to conduct air-scrubbing (supplying water amount: 3 m.sup.3 /Hr, supplying air amount: 5 Nm.sup.3 /Hr, air-scrubbing period: 5 min) with a valve on the filtrate side of the hollow fiber membrane module shut while feeding raw water.

Detailed Description Text (174):

After the air-scrubbing, the hollow fiber membrane module was again weighed. The weight was 103% of the initial weight.

Detailed Description Text (175):

This means that suspended solids were discharged by air-scrubbing in an amount corresponding to 12% of the weight of the hollow fiber membrane module.

Detailed Description Text (176):

Further, the above hollow fiber membrane module was subjected to leakage check. No leak was observed.

Detailed Description Text (177):

The above hollow fiber membrane module was dismantled to observe the state of bonded and fixed portions. It was confirmed that both the external and central portions of the hollow fiber membrane bundle were sufficiently filled up with the bonding agent.

Detailed Description Text (180):

Using the hollow fiber membrane module obtained in Example 5, the operation was conducted. As raw water, river surface water having turbidity of from 3 to 340 degrees (average: 120 degrees) and a fine particle diameter of from 2 to 130 .mu.m (medium value: 43 .mu.m) was used.

Detailed Description Text (181):

The filtration was conducted according to a cross flow filtration type operation, in which raw water was fed into a hollow fiber membrane module at a constant flow of 8.0 [1/min.multidot.module.multidot.25.degree. C.] so as to be a ratio of an amount of water filtered through the membrane to that of water circulating of 1/1, with a constant filtration flow, i.e., a filtrate amount of 4.0 [1/min.multidot.module.multidot.25.degree. C.].

Detailed Description Text (182):

The operation was performed by repeating a 10 minute filtration followed by conducting back wash with filtrate at a flow of 6.0 [1/min.multidot.module.multidot.25.degree. C.] and air-scrubbing with compressed air at a flow of 8 [Nm.sup.3 /hr.multidot.module.multidot.25.degree. C.] simultaneously for one minute. The turbidity of raw water was 120 degrees. The total filtrate permeating membrane during the filtration step was 40 liters. The amount of suspended solids accumulating was 4.1.

Detailed Description Text (184):

After operating for a total of 3 months, the trans-membrane pressure reached 1.3 times the initial value. Then, the module was taken out from the device to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 95% of that of unused membrane. When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane outer surface was negligible.

Detailed Description Text (187):

Using the hollow fiber membrane module obtained in Example 5, a filtration operation was conducted. As raw water, river surface water having turbidity of from 0.1 to 3 degrees (average: 1.2 degrees), a fine particle diameter of from 0.5 to 30 μm (medium value: 7 μm) and a temperature of 18.degree. C. was used.

Detailed Description Text (188):

The filtration was conducted using a cross flow filtration type operation, in which raw water was fed into a hollow fiber membrane module at a constant flow of 3.0 [m.sup.3 /hr.module.multidot.25.degree. C.] so as to be a ratio of an amount of water filtered through the membrane to that of water circulating of 1/1, with a constant filtration flow, i.e., a filtrate amount of 1.5 [m.sup.3 /hr.module.multidot.25.degree. C.].

Detailed Description Text (189):

The operation was performed by repeating a filtration cycle in which back wash with filtrate at a flow of 1.5 [m.sup.3 /hr.module.multidot.25.degree. C.] and air-scrubbing with compressed air at a flow of 2 [Nm.sup.3 /hr.module.multidot.25.degree. C.] were simultaneously conducted for two minutes after 30 minute filtration. The turbidity of raw water was 1.2 degrees. The total filtrate permeating membrane during the filtration step was 0.75 m.sup.3. The amount of suspended solids accumulating was 0.13.

Detailed Description Text (190):

After the operation was performed for 10 months under the above-mentioned operation conditions, the trans-membrane pressure was 1.2 times the initial value. Then, the module was taken out from the apparatus to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 96% of that of unused membrane. When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane outer surface was negligible.

Detailed Description Text (193):

Using the hollow fiber membrane module obtained in Example 5, a filtration operation was conducted. As raw water, river surface water having turbidity of from 0.1 to 3 degrees (average: 1.2 degrees), a fine particle diameter of from 0.5 to 30 μm (medium value: 7 μm) and a temperature of 18.degree. C. was used.

Detailed Description Text (194):

The filtration was conducted using a cross flow filtration type operation, in which raw water was fed into a hollow fiber membrane module at a constant flow of 3.0 [m.sup.3 /hr.module.multidot.25.degree. C.] to obtain a ratio of an amount of water filtered through the membrane to that of water circulating of 1/1, with a

constant filtration flow, i.e., a filtrate amount of 1.5 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.].

Detailed Description Text (195):

The operation was performed by repeating a filtration cycle in which after 30 minute filtration, back wash with filtrate at a flow of 1.5 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.] and air-scrubbing with compressed air at a flow of 2 [Nm.sup.3 /hr.multidot.module.multidot.25.degree. C.] were simultaneously conducted for 1 minute and a flushing with raw water was conducted at a flow of 2.5 [m.sup.3 /hr.multidot.module.multidot.25.degree. C.] for one minute. The turbidity of raw water was 1.2 degrees. The total filtrate permeating membrane during the filtration step was 0.75 m.sup.3. The amount of suspended solids accumulating was 0.13.

Detailed Description Text (196):

After the operation was performed for 5 months under the above-mentioned operation conditions, the trans-membrane pressure was 1.2 times the initial value. Then, the module was taken out from the device to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 95% of that of unused membrane. When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), the damage of the membrane surface was negligible.

Detailed Description Text (199):

An operation was performed in parallel with Example 14 under substantially the same conditions as described in Example 14 except that the hollow fiber membrane module obtained in Example 8 was used. After the operation was conducted for 20 days, the trans-membrane pressure was 3.5 times the initial value.

Detailed Description Text (201):

Further, after the above-mentioned membrane module was washed with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution until the recoverability was saturated, the flux amount of pure water was measured. It was 66% of the initial value.

Detailed Description Text (202):

Subsequently, the hollow fiber membrane module was dismantled and the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that about 75% of the membrane surface was rough and a part of the open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (205):

An operation was performed in parallel with Example 15 under substantially the same conditions as described in Example 15 except that the hollow fiber membrane module obtained in Example 8 was used.

Detailed Description Text (206):

The filtration pressure gradually increased as the filtration operation proceeded. The trans-membrane pressure reached 3 times the initial value in the second month and the fourth month of the filtration operation. Therefore, the module was subjected to chemical wash with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution.

Detailed Description Text (207):

When the total operation term was 5 months, the hollow fiber membrane module was taken out from the device to check the leakage. No leak was observed.

Detailed Description Text (208):

Further, after the above-mentioned membrane module was washed with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution until the recoverability was saturated, the flux amount of pure water was measured. It was 72% of the initial value.

Detailed Description Text (209):

Subsequently, the hollow fiber membrane module was dismantled and the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that about 70% of the membrane surface was rough and a part of the open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (212):

An operation was performed in parallel with Example 16 under substantially the same conditions as described in Example 16 except that the hollow fiber membrane module obtained in Example 9 was used.

Detailed Description Text (213):

After operating for 6 months, the trans-membrane pressure became 2.0 times the initial value. Judging that it would be impossible to continue the filtration operation further, the hollow fiber membrane module was dismantled. A single fiber of the hollow fiber membrane module was dismantled and subjected to chemical wash with a mixed solution of a sodium hypochlorite aqueous solution and a sodium hydroxide aqueous solution, and with a mixed solution of an oxalic acid aqueous solution and a nitric acid aqueous solution, and the flux amount of pure water of the single fiber was measured. It corresponded to 80% of that of an unused membrane. The outer surface of the membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that approximately 70% of the membrane surface was rough and a part of open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (216):

An operation was performed in parallel with Example 17 under substantially the same conditions as described in Example 17 except that the hollow fiber membrane module obtained in Example 9 was used.

Detailed Description Text (217):

After operating for 4 months, the trans-membrane pressure reached 2.0 times the initial value. Judging that it would be impossible to conduct the filtration operation further, the hollow fiber membrane module was dismantled. After a single fiber of the hollow fiber membrane module dismantled was subjected to chemical wash with a mixed solution of a sodium hypochlorite aqueous solution and a sodium hydroxide aqueous solution, and with a mixed solution of an oxalic acid aqueous solution and a nitric acid aqueous solution, the flux amount of pure water of the single fiber was measured. It corresponded to 82% of that of an unused membrane. The outer surface of membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that approximately 70% of the membrane surface was rough and a part of open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (220):

A filtration operation was conducted under substantially the same conditions as described in Example 18 except that the hollow fiber-membrane module obtained in Example 10 was used.

Detailed Description Text (222):

Subsequently, when the hollow fiber membrane module was taken out from the filtration operation apparatus and weighed, its weight was increased to 120% of the initial weight.

Detailed Description Text (223):

Again, this module was installed in the filtration operation device to conduct air-scrubbing (supplying water amount: 3 m.sup.3 /Hr, supplying air amount: 5 Nm.sup.3 /Hr, air-scrubbing period: 5 min) with a valve on the filtrate side of the hollow fiber membrane module shut while feeding raw water.

Detailed Description Text (224):

After the air-scrubbing, the hollow fiber membrane module was again weighed. The weight was 115% of the initial weight.

Detailed Description Text (225):

This means that suspended solids were discharged by air-scrubbing in an amount corresponding to 5% of the weight of the hollow fiber membrane module.

Detailed Description Text (226):

Further, the above hollow fiber membrane module was subjected to leakage check. No leak was observed.

Detailed Description Text (227):

The above hollow fiber membrane module was dismantled to observe the state of bonded and fixed portions. It was confirmed that a part of the central portion of the hollow fiber membrane bundle was not sufficiently filled up with the bonding agent.

Detailed Description Text (230):

The filtration operation was performed in parallel with Example 19 under substantially the same conditions as described in Example 19 except that the hollow fiber membrane module obtained in Example 8 was used.

Detailed Description Text (232):

After operating for a total of 3 months, the trans-membrane pressure reached 2.5 times the initial value. Then, the module was taken out from the device to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module after the operation was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution, and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 74% of that of an unused membrane.

Detailed Description Text (233):

When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), approximately 70% of the membrane surface was rough and a part of open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (236):

An operation was performed under substantially the same conditions as described in Examples 14 and 22 except that the hollow fiber membrane module obtained in Example 11 was used. After operating for 20 days, the trans-membrane pressure reached 2.9 times the initial value. After the filtration test, leakage was checked. No leak

was observed.

Detailed Description Text (237):

Further, the above-mentioned membrane module was washed with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution until the recoverability was saturated. When the flux amount of pure water was measured, it was 87% of the initial value.

Detailed Description Text (238):

Then, the outer surface of membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that approximately 20% of the membrane surface was rough and a part of open pores on the membrane surface was slightly covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (241):

An operation was performed in parallel with Examples 15 and 23 under substantially the same conditions as described in Examples 15 and 23 except that the hollow fiber membrane module obtained in Example 11 was used.

Detailed Description Text (242):

The filtration pressure gradually increased as the filtration operation proceeded. The trans-membrane pressure reached 3 times the initial value in the third month of the filtration operation. Therefore, the module was subjected to chemical wash with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution.

Detailed Description Text (243):

When the total operation term was 5 months, the hollow fiber membrane module was taken out from the device to check the leakage. No leak was observed.

Detailed Description Text (244):

Further, the above-mentioned membrane module was washed with sodium hydroxide aqueous solution, sodium hypochlorite aqueous solution, oxalic acid aqueous solution and nitric acid aqueous solution until the recoverability was saturated. When the flux amount of pure water was measured, it was 79% of the initial value.

Detailed Description Text (245):

Subsequently, the hollow fiber membrane module was dismantled and the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that about 20% of the membrane surface was rough and a part of the open pores on the membrane surface was covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (248):

An operation was performed under substantially the same conditions as described in Examples 14 and 22 except that the hollow fiber membrane module obtained in Example 12 was used and river surface water having a turbidity of 0.1 to 5 degrees (average: 2.2 degrees), a fine particle diameter of from 0.9 to 30 .mu.m (medium value: 9 .mu.m) and a temperature of 12 .degree. C. was employed as raw water. The turbidity of raw water was 2.2 degrees. The total amount of filtrate permeating the membrane during the filtration step was 0.43 m.sup.3. The amount of suspended solids accumulating was 0.13.

Detailed Description Text (249):

After operating for 8 months, the trans-membrane pressure reached 2.0 times the initial value. Judging that it would be impossible to conduct the filtration operation further, the hollow fiber membrane module was dismantled. A single fiber of the dismantled hollow fiber membrane module was subjected to chemical wash with

a mixed solution of a sodium hypochlorite aqueous solution and a sodium hydroxide aqueous solution, and with a mixed solution of an oxalic acid aqueous solution and a nitric acid aqueous solution. When the flux amount of pure water of the single fiber was measured, it corresponded to 83% of that of an unused membrane. The outer surface of membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that approximately 20% of the membrane surface was rough and a part of open pores on the membrane surface was slightly covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (252):

An operation was conducted in parallel with Examples 19 and 27 under substantially the same conditions as described in Examples 19 and 27 except that the hollow fiber membrane module obtained in Example 11 was used.

Detailed Description Text (254):

After operating for a total of 3 months, the trans-membrane pressure reached 2.0 times the initial value. Then, the module was taken out from the apparatus to check leakage. No leak was observed. Subsequently, the hollow fiber membrane module was dismantled and a single hollow fiber was subjected to chemical wash with a mixed solution of a sodium hypochlorite solution and a sodium hydroxide solution and a mixed solution of an oxalic acid solution and a nitric acid solution. When a pure water flux amount was measured, it corresponded to 78% of that of an unused membrane.

Detailed Description Text (255):

When the membrane outer surface of the hollow fiber membrane was observed with a scanning electronic microscope (magnification: 5,000 fold), approximately 20% of the membrane surface was rough and a part of open pores on the membrane surface was slightly covered. This was supposed to be a factor in causing a decrease of the water flux amount.

Detailed Description Text (258):

A filtration operation was performed at the same time of Example 28 under substantially the same conditions as described in Example 28 except that the hollow fiber membrane module obtained in Example 13 was used. After operating for 20 days, the trans-membrane pressure reached 3.3 times the initial value. After the filtration operation, leakage was checked. No leak was observed.

Detailed Description Text (259):

Further, the above-mentioned membrane module was washed with a sodium hydroxide aqueous solution, a sodium hypochlorite aqueous solution, an oxalic acid aqueous solution and a nitric acid aqueous solution until the recoverability was saturated. When the flux amount of pure water was measured, it was 70% of the initial value.

Detailed Description Text (260):

Then, the outer surface of membrane was observed with a scanning electronic microscope (magnification: 5,000 fold). It was observed that approximately 50% of the membrane surface was rough and a part of open pores on the membrane surface was slightly covered. This was supposed to be a factor in causing a decrease of water flux amount.

Detailed Description Text (261):

The results of Examples 1 to 32 are shown in Tables 2 to 9. As seen from Tables 2 to 9, it is apparent that in the purifying methods employing a hollow fiber membrane having an outer diameter of from 0.5 to 3.1 mm, one employing a wavy hollow fiber membrane, which is covered by the scope of the present invention, provides a more stable filtration than one employing a non-wavy hollow fiber membrane without waves. Further, it is seen that more preferable results can be obtained when the bulkiness is within the preferred range (1.45 to 2.00) or when

waves having different wavelength and wave height exist together.

Detailed Description Paragraph Equation (1):

Amount of suspended solids accumulating =(Raw water turbidity [degree]).times.
(Total amount of filtrate permeating membrane in filtration time [m.sup.3])/
(Membrane surface area [m.sup.2])

Detailed Description Paragraph Equation (4):

Packing ratio(%)=(Sectional area based on outer diameter of hollow fiber
membrane.times.Packing number of hollow fiber membrane per module).times.100/
(Sectional area based on inner wall of module case)

Detailed Description Paragraph Table (2):

TABLE 2 Membrane Inner Diameter Thickness Outer Diameter (mm) (mm) (mm) Flatness
Fiber Shape Bulkiness Example 1 0.7 0.28 1.25 0.968 Ununiform Wave 1.66 Example 2
0.7 0.28 1.25 0.969 Straight 1.43 Example 3 0.7 0.28 1.25 0.801 Uniform Wave 1.51
Example 4 0.7 0.28 1.25 0.948 Uniform Wave 1.44

Detailed Description Paragraph Table (3):

TABLE 3 Membrane Packing Number Packing Ratio Example 5 Example 1 300 36% Example 6
Example 1 1800 41% Example 7 Example 1 5760 40% Example 8 Example 2 300 36% Example
9 Example 2 1800 41% Example 10 Example 2 5760 40% Example 11 Example 3 300 36%
Example 12 Example 3 1800 41% Example 13 Example 4 300 36%

Current US Cross Reference Classification (1):

210/500.23

CLAIMS:

1. A hollow fiber membrane bundle which is prepared by collecting a plurality of wavy hollow fiber membranes so as to orient in a same direction with a bulkiness of from 1.45 to 2.00, the bulkiness being defined by $S1/S2$, where $S1$ is a cross sectional area of bundle of a given number of hollow fiber membranes and $S2$ is a cross sectional area of a single hollow fiber membrane multiplied by the given number of hollow fiber membranes, wherein the membrane has an inner diameter of from 0.3 to 1.7 mm, an outer diameter of from 0.5 to 3.1 mm, a membrane thickness of from 0.1 to 0.7 mm, and a flatness of from 0.8 to 1.0, where the flatness is a ratio of an inner minor axis to an inner major axis of the hollow fiber membrane.
2. The hollow fiber membrane bundle according to claim 1, wherein the flatness is from 0.9 to 1.0.
3. The hollow fiber membrane bundle according to claim 1, wherein the flatness is from 0.95 to 1.0.
4. The hollow fiber membrane bundle according to claim 1, wherein the membrane includes at least one material selected from the group consisting of polyethylene, polypropylene, polybutene, tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer (PFA), tetrafluoroethylene-hexafluoropropylene copolymer (FEP), tetrafluoroethylene-hexafluoropropylene-perfluoroalkylvinylether copolymer (EPE), ethylene-tetrafluoroethylene copolymer (ETFE), polychlorotrifluoroethylene (PCTFE), chlorotrifluoroethylene-ethylene copolymer (ECTFE), polyvinylidene fluoride (PVDF), polysulfone, polyether sulfone, polyether ketone, polyether ether ketone, polyphenylene sulfide, cellulose acetate, ethyl cellulose, polyacrylonitrile, and polyvinyl alcohol.
5. The hollow fiber membrane bundle according to claim 1, wherein the membrane has an average pore diameter of 0.001 to 1 .mu.m.
6. The hollow fiber membrane bundle according to claim 1, wherein the membrane has

an average pore diameter of 0.05 to 1 .mu.m.

7. The hollow fiber membrane bundle according to claim 1, wherein the membrane has a porosity of 50 to 90%.

8. A method for producing a hollow fiber membrane bundle comprising the steps of: i) extruding membrane production raw liquid in the form of a hollow fiber through a co-axial tube-in-orifice spinning nozzle to obtain a hollow fiber material, ii) cooling and solidifying or coagulating the hollow fiber material to obtain a hollow fiber membrane, and iii) collecting a plurality of the hollow fiber membranes so as to orient in the same direction; wherein the hollow fiber material is contacted by pulsation flow before or during the cooling and solidifying or coagulating step.

9. The method according to claim 8, wherein the membranes have a bulkiness of from 1.45 to 2.00, the bulkiness being defined by $S1/S2$, where $S1$ is a cross sectional area of bundle of a given number of hollow fiber membranes and $S2$ is a cross sectional area of a single hollow fiber membrane multiplied by the given number of hollow fiber membranes, and the membrane has an inner diameter of from 0.3 to 1.7 mm, an outer diameter of from 0.5 to 3.1 mm, a membrane thickness of from 0.1 to 0.7 mm, and a flatness of from 0.8 to 1.0, where the flatness is a ratio of an inner minor axis to an inner major axis of the hollow fiber membrane.

10. The method according to claim 8, wherein the membrane includes at least one material selected from the group consisting of polyethylene, polypropylene, polybutene, tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer (PFA), tetrafluoroethylene-hexafluoropropylene copolymer (FEP), tetrafluoroethylene-hexafluoropropylene-perfluoroalkylvinylether copolymer (EPE), ethylene-tetrafluoroethylene copolymer (ETFE), polychlorotrifluoroethylene (PCTFE), chlorotrifluoroethylene-ethylene copolymer (ECTFE), polyvinylidene fluoride (PVDF), polysulfone, polyether sulfone, polyether ketone, polyether ether ketone, polyphenylene sulfide, cellulose acetate, ethyl cellulose, polyacrylonitrile, and polyvinyl alcohol.

11. The method according to claim 8, wherein the membrane has an average pore diameter of 0.001 to 1 .mu.m.

12. The method according to claim 8, wherein the membrane has an average pore diameter of 0.05 to 1 .mu.m.

13. A hollow fiber membrane module, wherein a plurality of wavy hollow fiber membranes each having an inner diameter of 0.3 to 1.7 mm, an outer diameter of 0.5 to 3.1 mm, a membrane thickness of 0.1 to 0.7 mm and a flatness of 0.8 to 1.0, where the flatness is a ratio of an inner minor axis to an inner major axis of the hollow fiber membrane, is collected so as to orient in a same direction with a bulkiness of from 1.45 to 2.00, the bulkiness being defined by $S1/S2$, where $S1$ is a cross sectional area of a bundle of a given number of hollow fiber membranes and $S2$ is a cross sectional area of a single hollow fiber membrane multiplied by the given number of hollow fiber membranes, and mounted with a packing ratio of from 35 to 55%.

14. The hollow fiber membrane module according to claim 13, wherein the membrane includes at least one material selected from the group consisting of polyethylene, polypropylene, polybutene, tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer (PFA), tetrafluoroethylene-hexafluoropropylene copolymer (FEP), tetrafluoroethylene-hexafluoropropylene-perfluoroalkylvinylether copolymer (EPE), ethylene-tetrafluoroethylene copolymer (ETFE), polychlorotrifluoroethylene (PCTFE), chlorotrifluoroethylene-ethylene copolymer (ECTFE), polyvinylidene fluoride (PVDF), polysulfone, polyether sulfone, polyether ketone, polyether ether ketone, polyphenylene sulfide, cellulose acetate, ethyl cellulose, polyacrylonitrile, and polyvinyl alcohol.

15. The hollow fiber membrane module according to claim 13, wherein the membrane has an average pore diameter of 0.001 to 1 .mu.m.

16. The hollow fiber membrane module according to claim 13, wherein the membrane has an average pore diameter of 0.05 to 1 .mu.m.

17. The hollow fiber membrane module according to claim 13, wherein the membrane has a porosity of 50 to 90%.